



# **Investigation of Mechanical Properties of Graphene on Silicon Wafers**

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## ABSTRACT

Graphene is an atomically thin two-dimensional crystalline material with very low mass, high Young's modulus, high elastic strength, high optical transparency, high-electron mobility, high thermal conductivity and high degree of biocompatibility. Due to these extraordinary properties, graphene has many promising applications. Graphene can be synthesized in vastly different ways, for example by chemical vapour deposition and micromechanical exfoliation. However, the invariably poor graphene/substrate adhesion energy is a major drawback for ensuring the reliability, stability and longevity of sensors and other micro- and nano-mechanical devices, precluding us from achieving semiconductor technology requirements and rendering manufacturing efforts futile. Therefore, synthesising wafer level graphene that has sufficient quality and adhesion with the substrate is still an open and critical research problem.

To address these issues, we have demonstrated for the first time a fivefold improvement in adhesion between graphene and its underlying substrate, using a transfer-free, catalytic alloy approach for synthesising a monolayer of graphene on silicon carbide on silicon. An interfacial adhesion energy of  $5.7 \text{ J/m}^2$  between graphene and silicon carbide is found using double cantilever beam testing, as compared to  $1.02 \text{ J/m}^2$  reported for transferred graphene on silicon dioxide.

As the obtained adhesion energy is a good starting point for achieving reliable resonant sensors, we have fabricated and evaluated graphene coated silicon carbide membranes, showing quality factor ( $Q$ ) as high as  $2.7 \times 10^4$ . We have also investigated the influence of graphene coating on the quality factor of the silicon carbide membrane resonators and reported a significant reduction in damping when a graphene overlayer is present on silicon carbide membranes instead of a conventional metal layer.

## **CERTIFICATE OF ORIGINAL AUTHORSHIP**

I, Zulfiqar Hasan Khan, certify that the work presented in this thesis has not previously been submitted for a degree nor been submitted as part of the requirements for a degree except as fully acknowledged within the text.

I also certify that the thesis has been written by me. Any help that I have received in my research work and the preparation of the thesis itself has been acknowledged. In addition, I certify that all information sources and literature used are indicated in the thesis.

Production Note:  
Signature removed prior to publication.

Zulfiqar Hasan Khan

Sydney, Australia

August 2017

## **DEDICATION**

This thesis is dedicated to my family. Thank you for all of your love and support.

## ACKNOWLEDGEMENTS

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## THESIS FRAMEWORK

This thesis is divided into six chapters: Chapter 1: Introduction; Chapter 2: Published review paper–*Mechanical and electromechanical properties of graphene and their potential applications in Micro Electro-Mechanical Systems (MEMS)*; Chapter 3: Methodology; Chapters 4: Unpublished paper–*Ultrahigh adhesion of epitaxial graphene on SiC on silicon*; Chapter 5: Mechanical performance of graphene on cubic silicon carbide membrane resonators on silicon, and Chapter 6: Conclusion.

Chapter 1 is the introduction, and includes a brief discussion of the research background, motivation, importance and scope of the work. It also provides the thesis framework and the list of publications.

Chapter 2 is the literature review in the form of a published journal paper, which reviews the types of graphene, the mechanical properties of graphene such as elastic and fracture properties, its thermo-mechanical properties, as well as its tribology, adhesion and electromechanical properties. This chapter includes a comparative study of the adhesion of graphene on different substrates as measured by different tests. Moreover, this chapter provides a general overview of MEMS resonators; dynamic resonant sensing parameters, such as resonance frequency and quality factor; resonant sensing principles; electrical actuation and detection schemes; and transduction techniques. Lastly, this chapter discusses the potential of graphene in MEMS and examples of applications. Overall, the idea of this chapter is to present a review and explanation of the existing literature on the mechanical properties of graphene.

The overall experimental methodology is introduced in Chapter 3. It starts with graphene synthesis, followed by characterization techniques (for evaluating the graphene and the membranes resonators) and for performing the failure analysis after



mechanical fracture or delamination. Next, the chapter explains double cantilever beam (DCB) and four-point bending (FPB) test sample preparation, the microresonator fabrication stages, including photolithography, silicon carbide (SiC) and silicon (Si) etching. Subsequently, it covers the principles of DCB testing, FPB testing and the optical measurements of the mechanical properties ( $f$  and  $Q$ ) of membrane resonators by using Mach-Zehnder optical interferometry. Lastly, it specifies all the implemented instruments and their specifications. The intention of this chapter is to discuss the equipment, methods, and the calculations that were used throughout this work.

Chapter 4 presents the sample preparation for the four-point bending test and the double cantilever beam-bending test. Chapter 4 focuses on the measurement of the adhesion energy of nickel-copper (Ni-Cu) alloy mediated catalytic graphene on SiC on Si by the DCB test. This chapter also describes the failure analysis undertaken, in order to verify the location of the debonding path. This result of the DCB test is significant because of the fact that catalytic graphene offers fivefold improvement in adhesion compared to adhesion between graphene and silicon dioxide (SiO<sub>2</sub>).

Chapter 5 includes the results obtained from the simulation, fabrication, characterization and the measurement of the mechanical properties of the graphene coated SiC resonators, Ni-Cu coated SiC resonators and uncoated SiC resonators. Thus, the chapter reports the influence of graphene coating on mechanical properties of SiC membrane resonator. Moreover, the chapter demonstrates  $Q$ -factor improvement of graphene coated SiC membrane resonator by backside etching.

Finally, a general conclusion is given in chapter 6, which provides a summary and benefits of this work; and suggests ideas for future advances in this area.

## LIST OF ABBREVIATIONS

Abbreviations/Symbols	Full name
CVD	Chemical vapor deposition
AFM	Atomic force microscopy
Si	Silicon
SiC	Silicon carbide
3C-SiC	Cubic silicon carbide
SEM	Scanning electron microscopy
MEMS	Micro-electro-mechanical systems
FEM	Finite element modelling
$G$	Strain energy release rate
$G_c$	Critical strain energy release rate
$\Psi$	Mode mixity/ Phase angle
$P_c$	Critical load
$F$	Resonant frequency
$Q$	Quality factor
$E$	Young modulus
$E'$	In-plane strain modulus of the substrate
$P$	Density
$L$	Distance between outer and inner dwell pin
DRIE	Deep reactive ion etching
XPS	X-ray photoelectron spectroscopy
a.u.	Arbitrary unit
SiO <sub>2</sub>	Silicon dioxide
Si <sub>3</sub> N <sub>4</sub>	Silicon nitride

## JOURNAL PUBLICATIONS

- 1 **Khan Z H**, Kermany A R, Öchsner A, Iacopi F. “Mechanical and electromechanical properties of graphene and their potential application in MEMS”. *Journal of Physics D: Applied Physics*, 50(5):053003, 2017.

## AWARDS

1. First prize in 3-minute presentation at School of Electrical and Data Engineering, UTS.
2. Finalist in the HDR research showcase, 2017, for the Faculty of Engineering and Information Technologies.

## CONFERENCES AND WORKSHOPS

1. ANN ECR Workshop Griffith University, 2015, Goldcoast, Queensland, Australia.
2. Z. H. Khan *et al.*, 5<sup>th</sup> International Symposium on Graphene Devices, 2016, Brisbane, Australia.
3. Z. H. Khan *et al.*, SPIE Conference, 2016, San Diego, California, United States.

